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A study of the structural rearrangements accompanying room temperature recovery of high purity aluminum

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A STUDY OF THE STRUCTURAL REARRANGEMENTS
ACCOMPANYING ROOM TEMPERATURE RECOVERY
OF HIGH PURITY ALUMINUM

BY

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A

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ABSTRACT

X-ray methods (Laue back reflection and transmission) and etch pit studies were used in order to follow structural changes in the deformed metallic lattice during room temperature recovery. The accumulative evidence indicates that the deformed lattice undergoes subboundary movements and regrouping of fragments within a few days after stressing. On the basis of a superficial investigation, it was found that fast neutron bombardment increases the rate of room temperature recovery. Through the use of a new etchant, a finer substructure was revealed within the Lacombe subgrain.

Glide strain values were determined for the series investigated, and a new approach was attempted in the study of substructures using the Laue transmission method on a specially prepared single crystal specimen of foil thickness. A way of disclosing the internal detail within the etch pits was found through inking.

ACKNOWLEDGEMENTS

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I. INTRODUCTION

Many investigators have speculated on the possible existence of a finer structure within the usual grain structure, and through X-ray diffraction and metallographic techniques have revealed that such finer structures indeed exist.

The subject of recovery of stressed single crystals has been intensively studied, the emphasis being placed on the structural rearrangements resulting from a high temperature anneal. The aspects of room temperature recovery have been, to this day, almost completely neglected. The author has attempted to follow changes within the deformed metallic lattice during room temperature recovery by means of Laue back reflection and transmission methods, and through etch pit studies. Also, the effects of fast neutron irradiation on recovery have been superficially explored. It is hoped that the present investigation will shed some light on the effects of plastic deformation and the role of such mechanisms as polygonization and subgrain boundary migration within the recovery process.

II. REVIEW OF THE LITERATURE

A. Introduction

The traditional classification of restoration processes distinguishes between recovery and recrystallization. Recovery and recrystallization derive their driving force from the same source, the stored free energy of cold work. They are thus distinguished from grain growth, for which interfacial energy provides the driving force.

Recovery is classically defined as a change in the physical and mechanical properties of a cold worked metal when annealed, with no discernible change in the microstructure. But, such phenomena as polygonization and subgrain boundary migration, which occur under conditions traditionally considered as characteristic of recovery, have invalidated the classical definition.

Recrystallization is considered to be the formation of strain free nuclei which are subsequently capable of growing by consumption of the cold worked metal. After recrystallization is complete, an entirely new structure exists which is strain free, and is characterized by high angle boundaries between the recrystallized grains.

The term "polygonization", introduced by Orowan[1], was used by Cahn[2] to describe the following process, which occurred when single crystals were plastically deformed through bending, and subsequently annealed. An excess number of dislocations of one kind were more or less randomly arrayed on slip planes and then, upon annealing, they were rearranged into low angle boundaries perpendicular to the slip planes, forming what was known as "polygons" between the boundaries. These phenomena were noted by the form of asterism observed in Laue X-ray photographs. Immediately after bending, the Laue spots were elongated and continuous in nature. After polygonization the spots were still elongated but were divided into discrete areas. This change was due to the alignment of edge dislocations in the walls during annealing.

The appearance of a substructure in deformed and unannealed aluminum single crystals--as evidenced by fragmented Laue spots--called for revision of the concepts of polygonization and recovery in hard metals. Either polygonization occurred instantaneously and simultaneously with plastic deformation or subsequently, accompanying room temperature recovery.

It is well known that the passage of high energy neutrons through a crystalline solid will cause damage to the crystal lattice. This damage is the result of the interaction between the neutrons and the atoms in their respective lattice sites. Seitz[3] suggested that resoftening takes place by diffusion of vacancies to combine with dislocations to give rise to climb. This climb should assist in the dislocations forming polygon boundaries. Leighly et al[4] have used this explanation as their interpretation of the slowing down of the recrystallization rate in deformed aluminum single crystals. The injection of vacancies and interstitials by radiation damage should accelerate the rate of polygon boundary development and the growth of the polygons (polygonization).

The emphasis in this literature survey will be placed on polygonization, strictly from the point of view of recovery. Substructure studies, and the analysis of asterism in Laue photographs have been stressed accordingly in this review.

B. Evidence

The first investigation of the nature of polygonization was made by Crussard[5], who found that asterisms occurring in a Laue photograph taken on a deformed aluminum crystal remained in the same position in a photograph taken after annealing the crystal. However, the asterism was split into several separate spots with little or no blackening of the film between them. Laue photographs taken after successive anneals indicated that the spots decreased in number but became sharper and always occupied the area of previous asterisms. He named the phenomena "recrystallization in situ".

Prior to Crussard's work, an investigation was undertaken by Andrade and Chow[6] on the glide properties of particular body centered metals over a wide range of temperatures. Contrastingly, the breaking up of the asterism into discrete spots in sodium and potassium single crystals was attributed to recrystallization about certain preferential, probably high strained crystallites. This breakup of the Laue spots in deformed crystals after annealing was observed earlier in aluminum by Collins and Mathewson[7]. However, no fundamental attempt was made to explain the observed break up of Laue spots.

Crussard's work led Guinier and Tennevin[8] to investigate polygonization and develop a method for increasing the resolving power of X-ray patterns which detected structural changes that had previously been missed. Using this method to study polygonization of high purity aluminum single crystals, they concluded that it was normal for a cold worked metal to transform to the polygonized state during annealing, if lattice curvature is present, and recrystallization does not intervene. It was shown that the degree of deformation had a powerful influence on the recrystallization temperature but a small effect on the temperature at which polygonization first appears, which was found to be about 450°C.

This early work produced evidence that a deformed crystal is continuously bent and that discrete perfect blocks of quite large size are formed by the subsequent local straightening of the bent lattice ("polygonization"). Cahn[9] gives an excellent review of polygonization investigations up to 1950, and concluded that lattice curvature in crystals elongated in tension is due to deformation bands which were thought to be caused by inhomogeneous extension of the crystal. This view was supported by Crussard[5], who insisted that there is a relationship between the formation of subgrains or cells and the formation of deformation bands or "kinks", and that polygonization was responsible for their existence. Towner and Burger[10] found that subgrains existed in elongated crystals after deformation, particularly where lattice bending was most severe. From these investigations, it appears that polygonization or subgraining occurs during deformation, especially in regions of deformation bands or lattice curvature.

During the last thirty-five years there have been three main theories advanced to explain X-ray asterisms. Their origin has been variously attributed to local curvature of the slip plane, macroscopic curvature of the crystals or to fragmentation. The fragmentation theory attributes X-ray asterism to the formation of lattice fragments with very small differences in orientation between neighboring fragments, which if sufficiently small would make the asterism continuous. Wood[11], on the basis of extensive experiments on X-ray line broadening concluded that fragmentation occurs as a result of deformation.

Honeycombe's[12] X-ray evidence suggests that in the case of aluminum deformed at room temperature, a fragmentation of the crystals occurs. This theory (fragmentation) has been the most popular, and seems to depend on polygonization for the removal of lattice distortions. Honeycombe believes that the fragments are responsible for the intensity maxima in the asterisms, the diffuse background originating from distorted regions which join the fragments and allow the crystal to remain continuous although locally disoriented. In his support, polygonization has been observed to occur primarily in the diffuse regions of the asterisms, as would be expected if these regions were more severely bent than the rest of the crystal.

Wood and Rachinger[13] identified the blocks or fragments with the regions between the slip lines, and the crystallites with a fine structure between the blocks. They conceived the crystallites as a kind of debris of transitional orientations corresponding to a fine structure of the slip lines. They regarded such blocks as being formed simultaneously with deformation. However, they do not believe recovery takes place through polygonization, but through the progressive absorption of the finer crystallites into the larger blocks already formed.

In 1949, Yen and Hibbard[14] noticed a break up of the asterism in back reflection Laue photographs of bent and unannealed single crystals of high purity aluminum. The fragments of the spots appeared to be aligned in a specific pattern divided into two groups, one roughly parallel to the traces of the active slip planes and the other normal to it. They rationalized this discrete asterism, or subdividing of spots, as due to a process of fragmentation occuring during room temperature deformation. The fragmentation appeared to take place by rotating the crystallites in the glide lamellae about an axis normal to the slip plane, in addition to the usual type of bending about an axis lying in the slip plane and perpendicular to the slip direction.

In 1951, Heidenreich[15] used electron transmission through thin sections of high purity aluminum to follow recovery changes in the cold worked metal. He showed that immediately after cold working, high purity aluminum exhibits a subgrain or domain structure of the order of 1 to 2 microns in size. The domains were not produced by small deformations--of the order of a few percent--and experienced no growth or change in size of any consequence after months at room temperature. Heidenreich considered the observed recovery domains as an early stage of the process of polygonization. Honeycombe[12] found no indication of polygonization in aluminum single crystals only slightly deformed at room temperature. Several crystals were deformed small amounts--between 2% and 6% elongation--and allowed to remain at room temperature. It was found that the X-ray patterns showed intensity maxima when the exposure was made immediately after deformation. The pattern did not alter after two days at room temperature, and in the same time no change in the yield stress of the deformed crystals could be detected. Honeycombe's results are supported by the fact that Guinier and Tennevin[8] were unable to detect polygonization in aluminum at temperatures below 450°C.

In 1953, Hunter and Robinson[16] studied the subgrain structure of 99.995% aluminum by means of the electron microscope and oxide film replicas. These subgrains are about 10,000 angstroms in diameter. Starting with an equiaxed subgrain structure, increasing amounts of reduction by cold rolling brought about elongation of the subgrains, higher degrees of fragmentation and increasing amounts of slip within the grains. These structural changes are identical to those that ordinary grains undergo during cold working. With annealing, the fragment boundaries disappeared, and subgrains began to form in increasing numbers as atomic rearrangement occurred. Thus the subgrain appears as a fundamental unit by itself.

As a result of the experiments on substructure by Gervais, Norton and Grant[17], a theory of subgrain formation was proposed to bridge the gap between the fragmentation theory and that based on polygonization. X-ray and metallographic evidence indicated that polygonization was the main factor in subgrain formation, since most of the subgrain boundaries were formed by polygonization of the smoothly bent regions delineated by kinking. However, the fragmentation theory was considered applicable to subgrains due to kinking, these subgrain boundaries being very sharp from the very beginning of the deformation process. They concluded that subgrains must form as a consequence of deformation, and are not a main cause of deformation. Beck, Ricketts and Kelly[18] examined subgrains with the electron microscope in high purity cold rolled aluminum concluding that they were stable for several months at room temperature but grew upon annealing at 350°C. These authors also found that subgrain growth and softening both had similar kinetics, suggesting that recovery and polygonization are closely related.

Gay, Hirsch and Kelly[19] introduced a model to explain experimental results up to 1953. This model differs from previous particle models of the cold worked state, for the particles are not produced by fragmentation and the boundaries between the particles cannot be regarded either as normal inter-crystalline boundaries or polygon boundaries. In the present model, particles are considered to be regions of relatively small plastic curvature, while the boundaries are regions of large plastic curvature. So, in fact, a foam structure of particles is produced by the deformation; the particles are regions of low dislocation density separated by boundaries of high dislocation density. In effect, the boundaries of the particles are slip bands, and the particles themselves are considered to be the regions between the slip bands. In their view, recovery is due to a rearrangement of the dislocations in the boundaries to reduce the strain energy.

In 1955, Lambot, Vassamillet and DeJace[20] studied single crystals of high purity aluminum (99.99%) deformed by a few percent in tension, using a special X-ray diffraction technique. The method was capable of revealing essentially perfect subgrains whose size is of the order of tens of microns and whose mutual disorientation is at least of the order of one minute of arc. They showed that one part of the matrix is fragmented into domains possessing a high internal perfection while the rest gives a diffuse background in the Bragg reflections. For the purest aluminum used, the diffuse background disappeared progressively below about 450°C, and at about 550°C for 99.95% aluminum. Above these temperatures, the growth of certain nearly perfect subgrains took place.

In annealed grains or single crystals, a mosaic structure has long been established by the early X-ray work of Darwin and Bragg; the characteristics are a relative tilt of the elements from a few seconds to a few minutes of arc according to the degree of imperfection, and an approximate size of 10^{-4} cm. Lacombe and Beaujard[21] in their classic study of etch pits in high purity aluminum showed single crystals to be composed of an aggregate of little crystalline blocks, whose orientations are very slightly different, and whose dimensions are larger than those of the submicroscopic mosaic structure. Etching techniques have been very useful in the study of substructure; these techniques are, nevertheless, less sensitive than X-ray methods.

Livingston[22] used the double etch method to study dislocation distributions in as-grown, annealed and deformed crystals of 99.999% copper. Considering that the etchant will widen any pre-existing pit, but will only continue to deepen it as long as the dislocation responsible for it remains, the mobility of the dislocations could be well ascertained. Polygon walls, consisting of an array of edge dislocations in a plane perpendicular to their slip plane, were formed in a specimen by a prior deformation and high temperature anneal. An etch-stress-etch sequence on such a sample showed the motion and dissolution of polygon walls under low stress at room temperature. However, the subboundaries present in the as-grown crystals did not move, and it was found that these same original subboundaries were a serious obstacle to dislocation motion. According to Livingston, the dislocations in these boundaries are most probably not in simple parallel arrays with a single Burger's vector, nor will their Burger's vector in general be that of the most highly stressed slip systems, and therefore the immobility of these boundaries is not surprising. He concluded that it is possible that individual dislocations leave these boundaries at low stresses, but this event would be very difficult to detect by the double etch method.

In 1962, Wright[23] investigated the competition between recrystallization and polygonization in stretched and annealed single crystals of high purity (99.992%) aluminum. He concluded that there is a critical glide strain (about 0.17) below which polygonization will prevail in the annealed structure. Room temperature recovery prior to annealing seemed to favor polygonization of the metal.

In 1963, Szmid and Szarras[24] studied the effect of fast neutron bombardment on the mosaic structure of aluminum single crystals. The maximum neutron dose used was of the order of 5×10^{18} n/cm^2. The changes in mutual disorientation of the individual mosaic blocks or larger lattice fragments were recorded through the use of an oscillating film spectrometer. It was observed that many mosaic blocks, and even groups of blocks, were displaced during fast neutron irradiation. As a result of the irradiation, a certain amount of twisting of mosaic blocks relative to each other took place; the great majority of crystals tested evidenced a degree of twist within the zero to five minutes (of angle) range. They suggested that these changes were caused by a process of vacancy annihilation on the block boundaries, which are effective vacancy sinks. This process of vacancy annihilation, they proposed, could change the forces acting on the blocks, bringing about changes in mutual configuration of the microblocks.

Leighly, Perkins and McCune[4] investigated the recrystallization rate in 99.996% aluminum single crystals. They observed that a single crystal strained 10% in tension did not recrystallize upon heating in a goniometer furnace at 500°C- suggesting that the magnitude of the glide strain is of prime importance in determining whether recrystallization will take place. They subsequently found that holding at room temperature for long periods of time was sufficient to slow down or block recrystallization. To explain these observations they proposed a mechanism by which the excess number of vacancies above equilibrium concentration arranged themselves into polygon boundaries through the process of climb. These low angle polygon boundaries have been shown by Cottrell[25] to be a more stable dislocation configuration.

III. EXPERIMENTAL PROCEDURE

A. Production of High Purity Aluminum Single Crystals

The single crystals used in the experiment were produced by the strain anneal method as modified by Leighly and Perkins[26]. Generally, strain anneal single crystals have greater perfection than those grown from the melt. The Aluminum Company of America supplied the 99.992 per cent pure aluminum, in the form of cold rolled sheet, that was used as starting material. The chemical analysis supplied with the sheet is as follows:

Cu	0.005
Fe	0.002
Si	0.001
Al	99.992 (by difference)

Specimens measuring 0.5 x 4 inches were cut from the sheet having a nominal thickness of 0.050 inches. As in the work performed by Wright[23], all specimens were given a preliminary anneal at 640°C for 2 hours in order to remove any fabrication strains present. The specimens were not etched to reveal grain boundaries until the entire strain anneal process was completed, and the single crystal grown. Since metallographic examination of the single crystals was intended as part of the investigation, etching was kept to a minimum.

Leighly et al.[26] reported that critical strain was applied by wrapping the specimens around a 1 3/4 inch diameter cylinder and subsequently straightening against a flat surface. Wright[23] established his optimum diameter at 1 1/2 inches for a specimen thickness of 0.040 inches. As a result of the present investigation, an optimum diameter of 1 13/16 inches was established for a specimen thickness of 0.050 inches. It was observed that larger diameters - up to about 2 inches - would produce larger single crystals. But, invariably these large single crystals contained several island grains which would certainly influence the course of plastic deformation, and for this reason, had to be rejected. Smaller diameters gave smaller single crystals.

Annealing temperature was found to be very critical in the strain anneal method, the ideal being the highest temperature possible before the onset of melting. A temperature of 650°C for a period of two hours gave the best results.

Another important variable to consider is specimen thickness. Efforts to grow single crystals using the strain anneal method on specimens 0.0625 inches thick were largely wasted, as only a few single crystals could be grown and in most cases only after a second bending.

B. General Specimen Preparation

After undergoing the stress anneal sequence, the specimens were etched in Tucker's etchant (45% HCl, 15% HNO_3, 15% HF and 25% H_2O). Etching was accelerated by heating the solution to about 80°C; the entire specimen was submerged in the etchant, and the solution kept in constant motion by tilting the tray to either side. The presence of HF required that etching be carried out under a hood in a polyethylene tray.

The specimens selected for study by the Laue back reflection method received only a preliminary etching using Tucker's solution. Laue back reflection photographs were taken of the specimens before deformation, immediately after deformation, and at frequent intervals thereafter until changes in the Laue spots were evident. Copper white radiation from a Norelco X-ray unit operated at a tube voltage of 30 KV and a tube current of 15 ma was used. With a 3 centimeter specimen to film distance, exposure times of around 3 hours were required, depending on the single crystal orientation. The specimens were affixed to a special mount with apiezon wax, and visually positioned. Tests were discontinued at the slightest evidence of specimen movement. The experimental set up is shown in Figure 1.

The crystals were oriented from their Laue back reflection patterns using the method first described by Grenninger[27]. This is a classical technique and is described

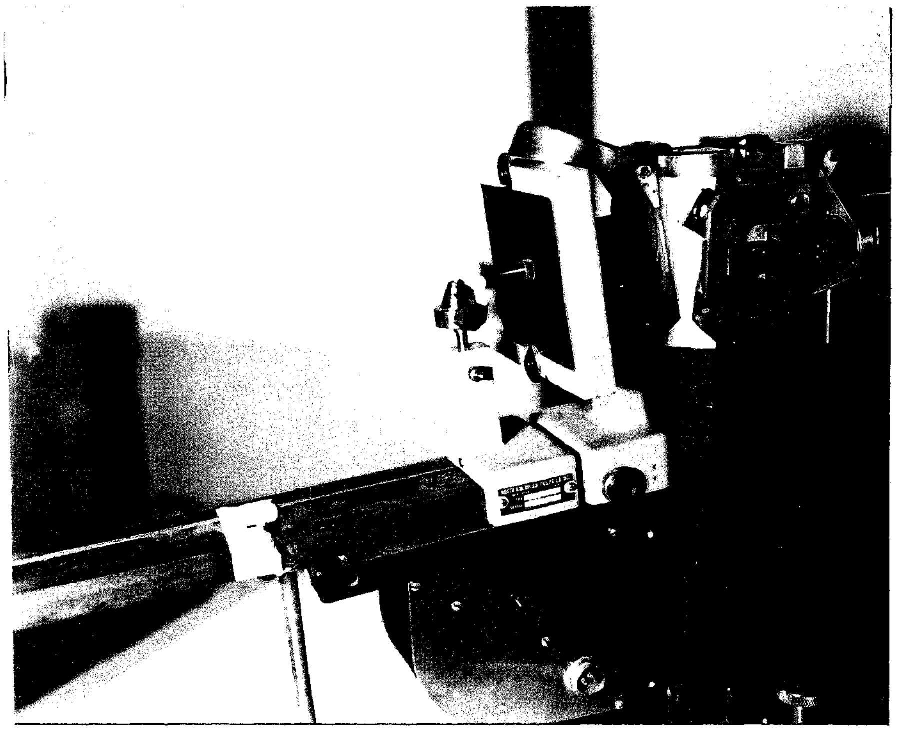

Figure 1

Experimental set up for the Laue back reflection, with radiation shield removed.

in detail in standard texts on X-ray diffraction such as Cullity[28]. When the orientation of a single crystal is completed, the results consist of poles or normals to crystallographic planes plotted on a stereographic projection or Wulff net. The poles on this type of projection can be identified and assigned Miller indices from their angular relationship as tabulated in Cullity[28]. After the identification of any 3 poles, all other poles can be plotted from symmetrical considerations.

The specimens that were to be examined metallographically were chemically polished, the solution being heated to 75°C for a more efficient dissolving action. The composition of this chemical polishing solution is as follows:

70% Orthophosphoric acid

15% Nitric acid

15% Distilled water

C. Glide Strain Determination

The stereographic projections for each crystal were used to locate the position of the tensile axis on a (001) standard projection. From this type of projection, the first slip system to become operative during plastic deformation may be determined as described by Schmid and Boas[29]. Also, the angles between the slip plane and the tensile axis, and those between the slip direction and the tensile axis may be measured. The (001) standard projection showing the location of the tensile axis of all specimens is shown in Figure 2. When the angles between the tensile axis and the slip plane and slip direction respectively are known, the glide strain for each elongation can be calculated. Glide strain is described by Schmid and Boas[29] as the relative displacement of two glide planes of unit distance from each other, and is calculated by the formula:

$$S = \frac{1}{\sin x_o} \left(\sqrt{D^2 - \sin^2 \lambda_o} - \cos \lambda_o \right)$$

where S = glide strain

x_o = angle between the glide plane and tensile axis

λ_o = angle between glide direction and tensile axis

$D = 1 + L_1/L_o$

L_1 = length after extension

L_o = length before extension

Using the above formula the glide strain for each specimen was calculated, including that of specimen B, whose tensile axis is favorably positioned to undergo double slip in the (001) standard projection (Fig. 2). This was done for the sake of simplicity.

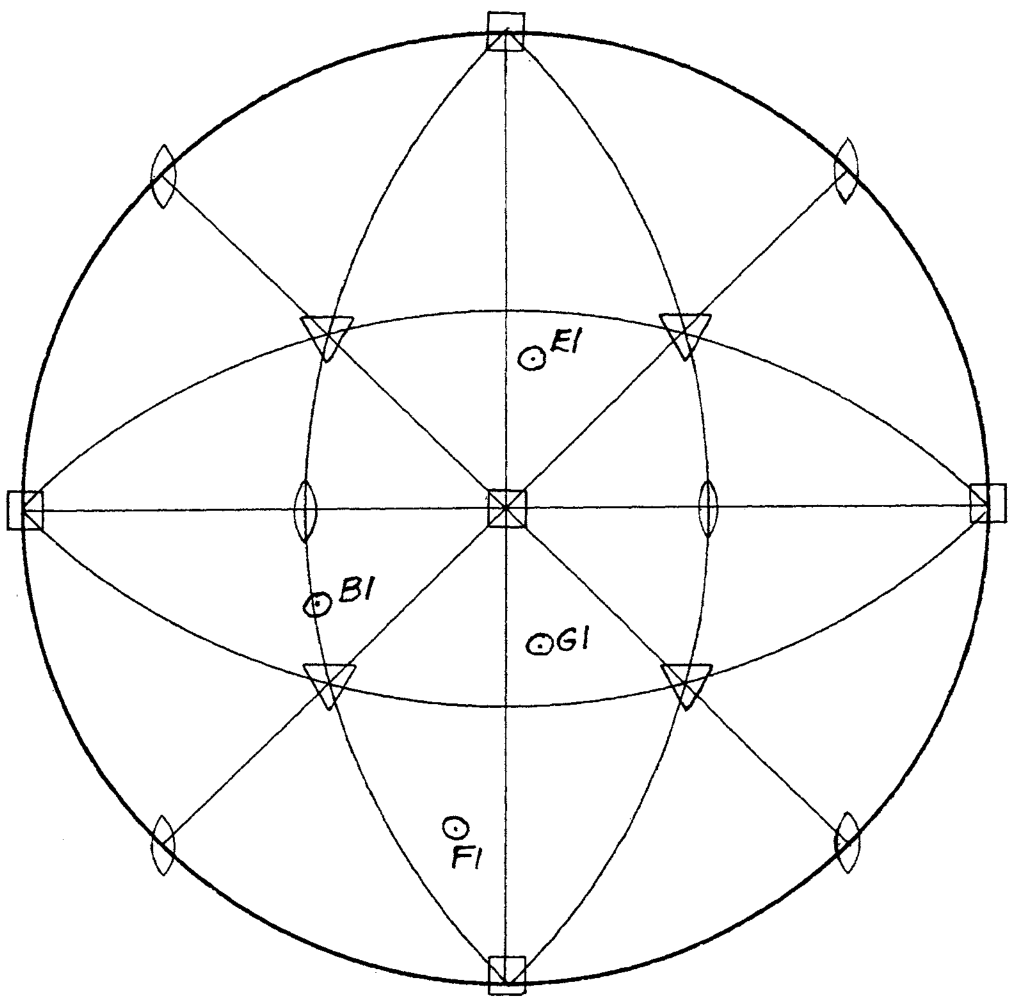

Figure 2

(001) Standard projection showing the location of the tensile axis of each specimen before deformation.

IV. EXPERIMENTAL RESULTS AND DISCUSSION

A. Back Reflection Laue Method

The results of applying this X-ray method to the investigation of room temperature recovery are summarized in Tables 1 and 2. Four single crystals were stressed in increasing amounts, and the Laue photographs taken of these specimens during the stress-recovery cycle were grouped into four corresponding series. These series have been designated by capital letters, and the photographs within each series are differentiated by numbers.

1. Discussion on "E" Series

E1 (Fig. 3) shows the characteristic well defined, rounded spots of the unstressed single crystal. E2 (Fig. 4), taken immediately after a 3.3% tensile elongation shows elongated spots, some in the direction of zone lines, others normal to them. After 4 days at room temperature, photograph E3 (Fig. 5) shows no appreciable change of the deformed Laue spots. However, after 6 days of recovery, some of the spots have been replaced by two identical subspots, sharply defined, as shown by E4 (Fig. 6). Obviously, the deformation was not of a magnitude sufficient to produce fragmentation and the formation of subspots simultaneously with stressing. The degree of perfection and sharpness of these subspots, and the fact that an original Laue spot has split into only two, and identical, subspots can be tied to the very small value of glide strain for this series.

SERIES	TREATMENT	RECOVERY TIME (HR.)	EXPOSURE LAUE (HR.)
E1	Unstressed	---	3.5
E2	3.3% Tensile Elong.	0	3.8
E3	No further	96	3.6
E4	No further	144	3.5
E Series Glide Strain - - - - - - - - - - 0.073			
B1	Unstressed	---	3.2
B2	5% Tensile Elong.	18	3.9
B3	No further	44	3.1
B4	No further	65	3.1
B5	No further	72	2.9
B6	No further	264	3.4
B Series Glide Strain - - - - - - - - - - 0.086			
F1	Unstressed	---	2.6
F2	10% Tensile Elong.	0	3.4
F3	No further	24	3.9
F4	No further	48	4.3
F5	No further	48	1.6
F Series Glide Strain - - - - - - - - - - 0.175			

TABLE I
Experimental data on Laue back reflection photographs.

I. LAUE BACK REFLECTION

SERIES	TREATMENT	RECOVERY TIME (HR.)	EXPOSURE LAUE (HR.)
G1	Unstressed	---	2.7
G2	10% Tensile Elong.	0	3.0
GU1	No further	5	3.4
GU2	Annealed 1 hr. (330°C)	53	3.5
GI1	Irradiated (5 hr., 10KV)*	33	3.5
GI2	Annealed 1 hr. (330°C)	58	3.7

G Series Glide Strain - - - - - - - - - - - - 0.209

*equals a total flux of approximately 3 x 10^{14} neutrons/cm^2

II. LAUE TRANSMISSION

SERIES	TREATMENT	RECOVERY TIME (HR.)	EXPOSURE LAUE (HR.)
L1	Stressed	0	0.6
L2	No further	120	0.6

TABLE II
Results of special substructure investigation.

Figure 3
Laue back reflection photograph of unstressed E specimen

Figure 4
Laue back reflection photograph of same specimen E, immediately after a 3.3% tensile elongation. Notice smeared Laue spots.

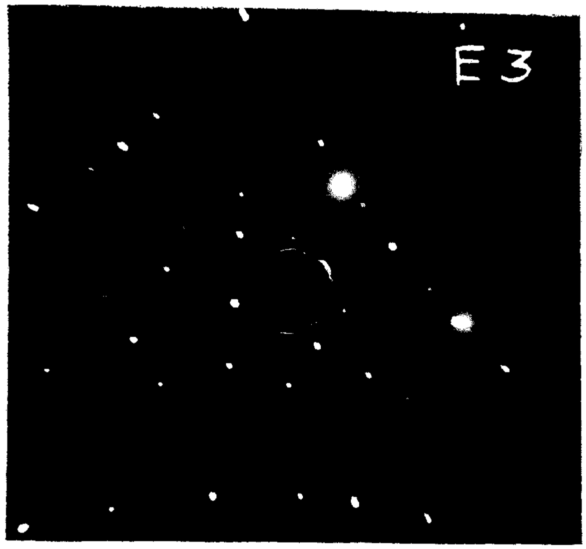

Figure 5
Laue back reflection photograph of stressed specimen E, after 4 days at room temperature. No appreciable changes in the Laue spots are evidenced.

Figure 6
Laue back reflection photograph of stressed specimen E, showing sharp subspots after 6 days of room temperature recovery.

2. Discussion on "B" Series

As before, B1 (Fig. 7) shows the Laue pattern for the unstressed single crystal. Eighteen hours after a 5% tensile elongation, B2 (Fig. 8) shows that fragmentation has taken place. Some of the original spots have split into three subspots, others into just two subspots, but their boundaries remain as yet undistinct. These subspots are unequal in size and intensity, the first and outermost one being the most intense in B2.

Photographs B3 (Fig. 9) and B4 (Fig. 10) record the process of room temperature recovery, manifested by a change in the intensity of the subspots. The subspots become well defined with time and tend towards a uniform intensity, that of the most intense subspot present. These subspots tend to consolidate in number, and it is hard to find in photograph B3 more than two subspots. Intensity changes are accompanied by changes in spacing between subspots, for it follows that as the fragment boundaries become increasingly defined, the spacing between subspots should increase in a corresponding manner.

When comparing B3 and B4, and in particular the lower section of each photograph next to the edge, where two equal subspots are joined by a diffuse area, it is seen that as the two outer subspots tend towards equal intensity, this diffuse area becomes well defined. A fine structure appears, striated under a magnifying glass, but which does not attain

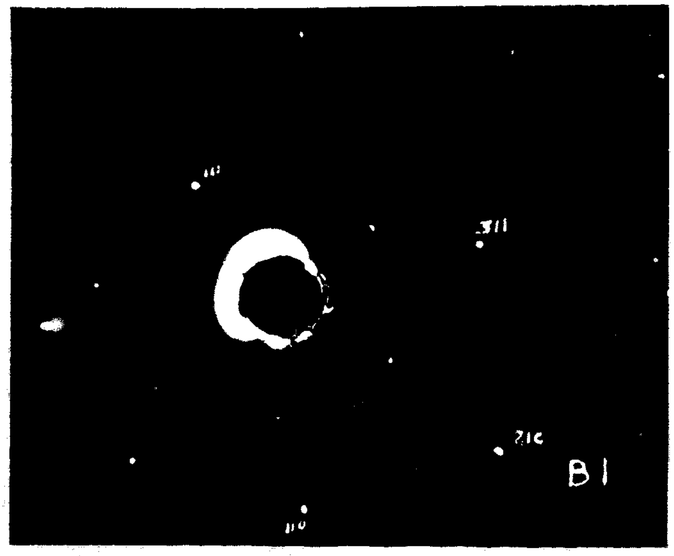

Figure 7
Laue back reflection photograph of the unstressed specimen B.

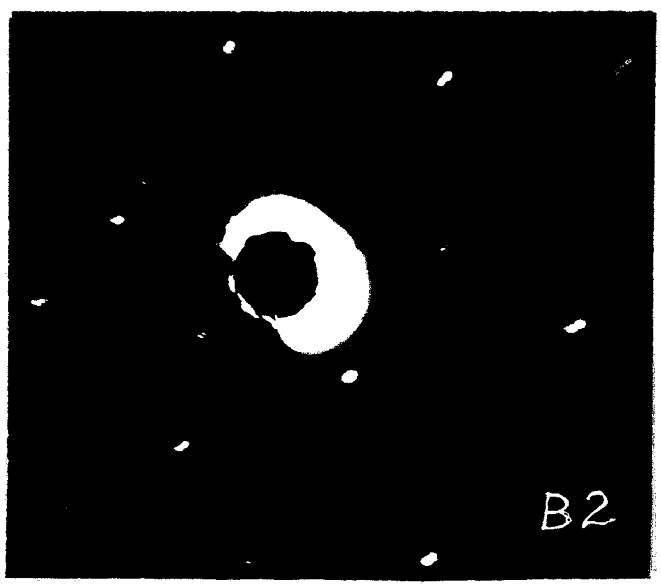

Figure 8
Laue back reflection photograph of same specimen B, elongated 5% in tension, and held for a period of 18 hours at room temperature.

Figure 9
Laue back reflection photograph of stressed specimen B, after 2 days at room temperature; recovery being manifested by changes in the relative intensity of subspots with time.

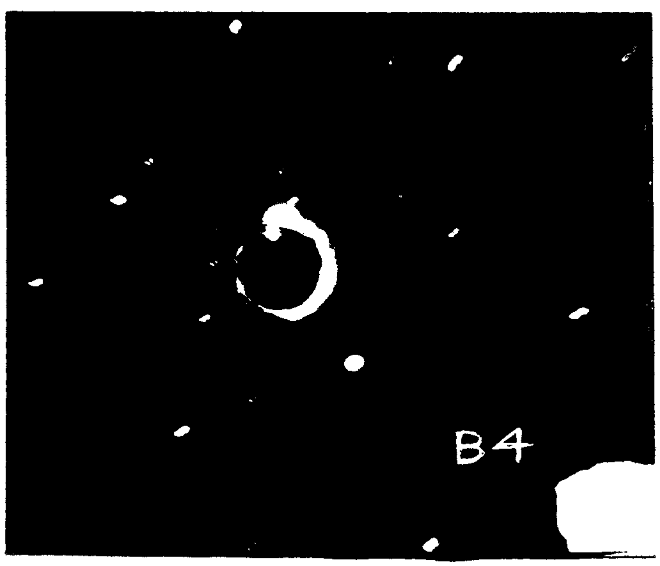

Figure 10
Laue back reflection photograph of stressed specimen B. Notice further consolidation and sharpening of subspots with time.

the intensity of the two outer subspots.

Because the single crystal undergoes inhomogeneous deformation, it is important that the same area on the specimen be irradiated by the X-rays when taking a Laue pattern. The appearance of Photograph B5 (Fig. 11) is significantly different to that of B4. The X-ray beam was shifted a small amount from the original spot, causing the apparent contrast. However, the effects of room temperature recovery on this new area were found to be similar to the previously observed changes. A period of eight days separate photographs B5 and B6 (Fig. 12), the subspots appearing quite distinct in B6.

In this case, the glide strain is still small enough so that the Laue spots are only slightly smeared, occupying a small angular spread of photograph and eventually evolving into just a pair of subspots.

3. Discussion on "F" Series

F1 (Fig. 13) shows the Laue photograph corresponding to the annealed, perfect state. F2 (Fig. 14) taken immediately after a 10% tensile elongation shows that the original Laue spots have been extended in two directions - in the same direction as the zone lines, and also normal to them. The deformation has caused the original spots to break up into two sizable fragments or coarse subspots. These subspots are very poorly defined and cover a larger area of photograph

Figure 11
Laue back reflection photograph of stressed specimen B, but of a different area (due to slight shift in the X-ray beam). No subspots are present after 3 days of room temperature recovery.

Figure 12
Laue back reflection photograph of same area of specimen B as shown in Fig. 11, but taken 8 days later. Subspots are quite evident.

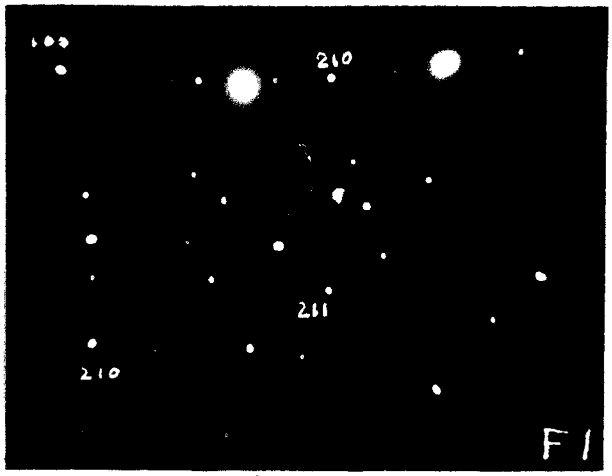

Figure 13
Laue back reflection photograph of unstressed specimen F.

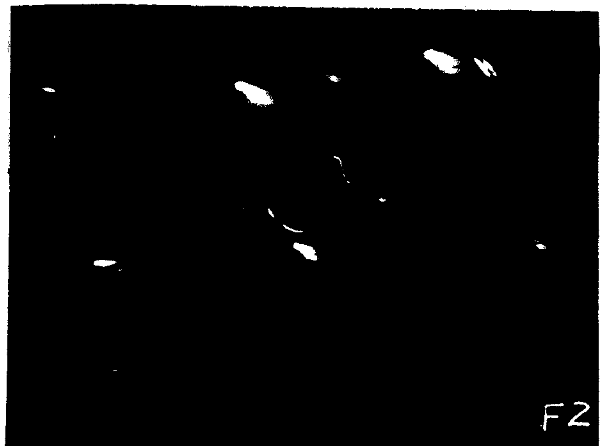

Figure 14
Laue back reflection photograph of same specimen F, showing coarse subspots formed immediately after a 10% tensile elongation.

Figure 15
Laue back reflection photograph of stressed specimen F, showing changes in relative intensity of subspots after 1 day at room temperature.

than those of Series E and B.

There is also an observable intensity difference between the two subspots. Photograph F3 (Fig. 15) taken after 1 day at room temperature shows a change in the relative intensity of the two-subspots - the least intense subspot in Photograph F2 now appears as the more intense of the pair. These changes seem to point out that the deformed lattice, through the process of recovery, undergoes subboundary movements and regrouping of fragments in its search for a lower energy configuration.

Even though Photograph F3 has a half hour longer exposure than F2, it should not affect the relative intensity of the subspot pair. This can be readily seen by comparing Photographs F4 (Fig. 16) and F5 (Fig. 17) taken of the same specimen under the same conditions, after two days at room temperature, and differing in exposure time by 2.7 hours. By this comparison, it is easily confirmed that the most intense subspot in Photograph F4 remains as the most intense of the pair in F5.

In this case, the glide strain was large enough to cause a larger angular spread of Laue spot. The nature of the deformation was such that the subspots are unequal in size, have no distinct, straight boundaries, indicative of the existence of a finer substructure within the subspots.

Figure 16
Laue back reflection photograph of stressed specimen F, after a 2 day recovery period; taken with a longer Laue exposure (4.3 hours) for the sake of comparison.

Figure 17
Laue back reflection photograph of stressed specimen F; taken of the same area, and identical with Fig. 16 but for a shorter Laue exposure (1.6 hours).

4. Discussion on "G" Series

The effect of fast neutron irradiation on room temperature recovery of deformed high purity aluminum single crystals was superficially explored.

A back reflection Laue photograph of the unstressed single crystal - taken of a spot at the center of the specimen - is shown in Photograph G1 (Fig. 18). Photograph G2 (Fig. 19) taken right after a 10% tensile elongation shows smeared spots broken up into a great many fragments or subspots, a few of them quite clearly defined.

Next, the single crystal was sectioned into two equal segments using a jeweler's saw, and the abraded edges were submerged in Tucker's etchant for relief of mechanical stresses introduced. One of the segments was taken to the nuclear reactor to be irradiated (GI), and the other segment was kept as a control at room temperature (GU). While one of the segments was being irradiated, Laue photographs were taken of a spot at the center of the control specimen with time. Photograph GU1 (Fig. 20) taken after 5 hours at room temperature, when compared to G2 shows changes even after a small recovery time.- the fact that different spots of the same specimen are being compared has to be taken into account.

The irradiated segment (GI) was kept inside the reactor for 5 hours at 10 KW. Photograph GI1 (Fig. 21), taken 33

Figure 18
Laue back reflection photograph taken of an area at the center of the unstressed specimen G.

Figure 19
Laue back reflection photograph of the same specimen G, showing subspots formed immediately after a 10% tensile elongation.

Figure 20
Laue back reflection photograph of an area at the center of control specimen GU, after 5 hours at room temperature.

Figure 21
Laue back reflection photograph of the irradiated (5 hours in the reactor at a power level of 10 KW) segment GI, after 33 hours at room temperature.

hours after stressing, when compared with both G2 and GU1 shows that radiation damage has brought about a still greater amount of recovery. (This last observation seems to confirm the mechanism proposed by Leighly, Perkins, and McCune[4] for the formation of polygon walls by dislocation climb). The regularity of the assemblage is altogether remarkable.

Next, identical corners were clipped from each segment to induce recrystallization, and both segments were given a low temperature anneal (330°C) for a period of one hour. This temperature was chosen to verify Guinier and Tennevin[8]'s experimental statement that up to a temperature of 450°C annealing leads to no perceptible alteration in the focused Laue spot. Contrary to their results, changes in the spots are quite evident in Photographs GU2 (Fig. 22) and GI2 (Fig. 23) taken after the low temperature anneal.

Photograph GI2, corresponding to the irradiated segment shows that after annealing, a much finer substructure is present. The coarse fragment pairs originally composing each subspot have been replaced by "comet streaks" upon annealing. These "comet streaks" are characterized by a main subspot or fragment, which makes up the actual body of the comet, followed by a "nebulous tail" of fragments so fine and of almost equal inclinations as to give the impression of continuity. This is indeed an advanced stage of recovery.

Figure 22
Laue back reflection photograph of an area at the center of control specimen GU, after annealing for an hour at 330°C.

Figure 23
Laue back reflection photograph of an area at the center of the irradiated segment GI, after annealing for an hour at 330°C.

Photograph GU2 taken of the control segment after annealing also shows the observed "comet streaks" of photograph GI2, but in this case there is no clear cut demarcation between the body of the "comet" and its "tail". Sizable fragments of quite different inclinations, and very definite spacings are still found in the tapered end of the "comet's tail". There is no gradual refinement of the structure as was observed in GI2. Quite clearly, recovery has not progressed to the degree observed in the irradiated segment.

The two segments, following the anneal were etched with Tucker's etchant to reveal the presence of any recrystallized grains growing from the clipped corners. However, it was found that no new grains were formed, polygonization having prevailed over recrystallization. This seems to coincide with the observations by Leighly et al[30] which indicated that increased recovery time at room temperature retarded recrystallization. Wright[23], nonetheless found that some specimens which were well polygonized after a six month recovery period recrystallized in spite of their partial recovery when annealed at 640°C. His opinion was that the recrystallization temperature is increased by ambient temperature recovery if the glide strain is above the critical value (0.17). The glide strain was above critical value in this case, but the annealing temperature (330°C) was not high enough to test Wright's assertion.

B. Laue Transmission Method

1. Discussion on Experimental Approach

Transmission and back reflection Laue patterns made from the same deformed region usually differ markedly in appearance. Both show elongated spots, which are evidence of lattice bending, but the spots are elongated primarily in a radial direction on the transmission pattern while on the back reflection pattern they tend to follow zone lines. The shape of a back reflection spot is more directly related to the nature of the lattice distortion than is the shape of the transmission spot since, in the general case, circular motion of the end of the reflecting plane normal causes circular motion of the backward reflected beam, but elliptical motion of the forward reflected beam. For this reason, the back reflection method is generally preferable for studies of lattice distortion.

The question as to whether the observed changes in the Laue back reflection photographs of the stressed specimens were actual signs of room temperature recovery, and not the result of minute shifts in the X-ray beam, led to a new approach to ascertain the authenticity of the previous observations. This new approach envisioned Laue transmission photographs of single crystals of foil thickness. The single crystal, by progressive etching in Tucker's etchant was reduced in thickness from 0.050 inches to approximately 0.007 inches. By means of a pin, a hole 0.8 mm in diameter was made through a small piece of lead foil of approximately 0.010 inches in thickness; next, the piece was cemented to the specimen.

The same experimental set up as for the Laue back reflection method was used, just modified for the transmission method. With a specimen to film distance of 3.3 centimeters, an exposure time of 0.6 hours was maintained. Because the single crystal was of foil thickness and very hard to handle without cold working it, and because of the impossibility of applying tension to it any other way, the foil specimen was manually stretched. Since the specimen was so difficult to handle, the per cent elongation was not determined. The specimen was easily sectioned to a length of 7/8 inches for ease of handling and placed on the mount with apiezon wax, as before.

The method is very advantageous because no matter if the beam is shifted, the lead foil will prevent irradiation on any other area but the open area of the aperture. Also, there is no need to disturb the specimen during loading and unloading of film holders because of the special geometry of the transmission set up. A photograph of the single crystal used is shown in Fig. 24.

2. Discussion on "L" Series

L1 (Fig. 25) taken immediately after stressing shows elongated spots of different shapes; some of them being made

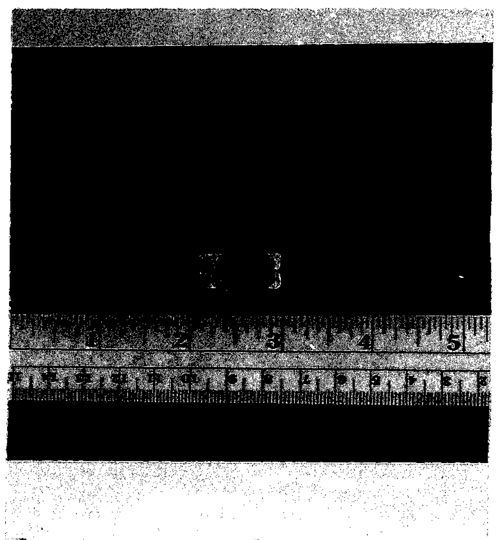

Figure 24
Photograph showing thin single crystal with cemented piece of lead foil, used with the Laue transmission method.

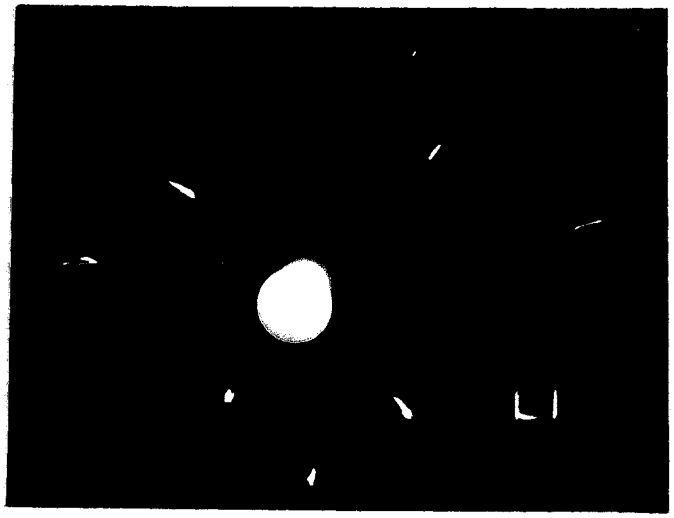

Figure 25
Laue transmission photograph of thin film specimen L immediately after stressing.

Figure 26
Laue transmission photograph taken after 5 days at room temperature. Notice vanished subspots.

up of several fragments or subspots of differing intensities. No further changes in the appearance of the subspots are evidenced until the fifth day of recovery. Photograph L2 (Fig. 26) taken on the fifth day shows definite alterations as pointed out by arrows A and B. Only a single subspot remains of the two originally present in photograph L1, as indicated by arrow A. Also, the lower section of subspot is missing in L2 as shown by arrow B. Some of the subspots which appear more or less faded in L1 have completely disappeared in L2. The results of L series prove that the deformed metallic lattice is subject to structural rearrangements even at room temperature, and within a few days of stressing. No further changes were evidenced afterwards, even though photographs were taken up until the twenty-fifth day after stressing.

C. Etch Pit Method

1. Experimental Evaluation

Early studies on etch pit techniques in order to follow subgrain boundary movements during room temperature recovery of deformed aluminum single crystals were very disappointing, and that is why the experimental emphasis was placed on the Laue method. For one thing, the unstressed single crystals grown by the strain anneal method had very high dislocation densities as shown by the great number of etch pits already assembled into low angle boundaries in Figure 27. This specimen was etched with Lacombe and Beaujards'[21] reagent as modified by Gervais, Norton and Grant[17]. The chemical composition of this etching reagent is as follows:

Methyl Alcohol	50cc
Hydrochloric Acid	32cc
Nitric Acid	50cc
Hydrofluoric Acid	2cc

The reagent was cooled in an ice bath during preparation, and a fresh solution made each time.

This same specimen was given a 10% tensile elongation and re-etched, in accordance with the "double etch method." The very high dislocation densities introduced by plastic deformation hindered the required observations. Also, the high reflectivity of the metallic surface made the study of the bottom of etch pits quite difficult. This difficulty was overcome by swabbing the specimen surface with plain

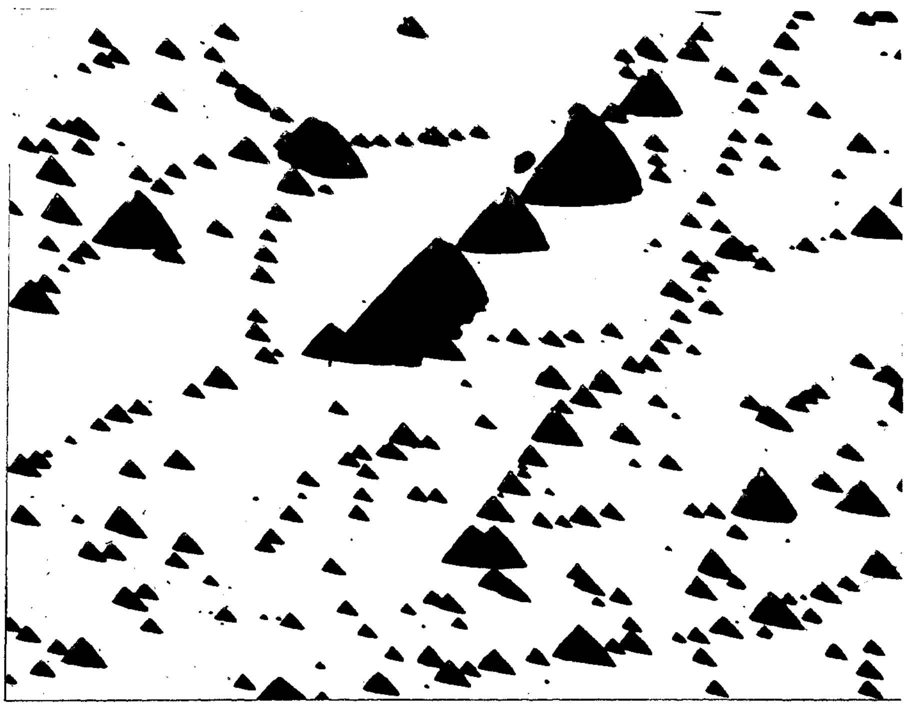

Figure 27
Unstressed single crystal etched with the modified Lacombe and Beaujard reagent. Visible subgrains are present. 500 X, bright field.

ink so that the internal detail of the etch pits was revealed. Figures 28 and 29 are photographs of the same area of inked specimen at different magnifications showing flat bottoms of moved dislocations and sharp bottoms of new or stable dislocations. Further etching with time gave no evidence of subboundary movements due to recovery. This does not necessarily mean, however, that dislocation rearrangements do not take place but rather that the double etch method has only limited applicability. This conclusion is supported by Livingston's[22] work on copper and by studies of dislocation pinning carried out by Stein and Low[31].

A great number of single crystals were studied using etch pit techniques, but only those specimens that gave triangular etch pits with the modified Lacombe and Beaujard etchant were useful. These triangular etch pits are found in aluminum single crystals having their surfaces nearly parallel to the (111) plane. The strain anneal method mostly produced single crystals giving square etch pits - surfaces parallel to (100) plane - upon etching. These single crystals exhibited a very anomalous behavior upon etching; some etched too fast, and others very slowly.

A finer structure within Lacombe's subgranular blocks was revealed by a second etching with another reagent (150 gms. $CuSO_4 \cdot 5H_2O$, 27cc H_2SO_4, 50 cc Ethyl Alcohol and 1000 cc of H_2O) after a preliminary etching with the modified Lacombe and Beaujard etchant. Etch pits of a different kind

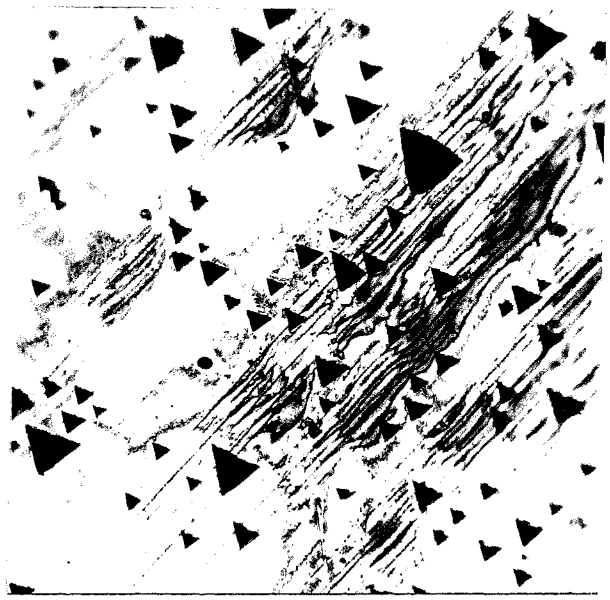

Figure 28
Same single crystal etched a second time with the modified Lacombe and Beaujard etchant to illustrate "double etch" method. Surface inked to reveal bottoms of etch pits.
250 X, bright field.

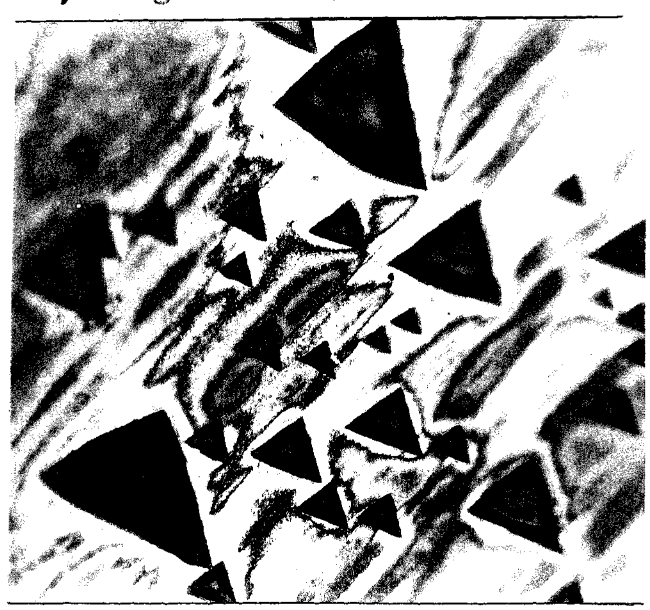

Figure 29
Same spot as in Fig. 28, but higher magnification. Note some of the etch pits have flat bottoms, others sharp bottoms.
500 X, bright field.

were formed along slip lines, migrating to form glide polygons within the Lacombe subgrain. It is quite possible that these new etch pits, the direct result of plastic deformation, have a greater bearing on the structure of the Laue subspot than the larger Lacombe etch pit. Figure 30 shows the fine structure within the Lacombe subgrain. It is interesting to recall that fine striations were observed in the course of this investigation within Laue subspots. These striations were also reported by Lambot, Vassamillet and DeJace[20] and interpreted as slip lines by Gay, Hirsch and Kelly[19] in their "foam" model of subgraining. Honeycombe[12] believed that the fine structure in asterisms from crystals deformed at room temperature might be attributed to the partial occurrence of polygonization. However, it is extremely difficult to attempt to correlate etch pit structures with the structures revealed by X-ray methods.

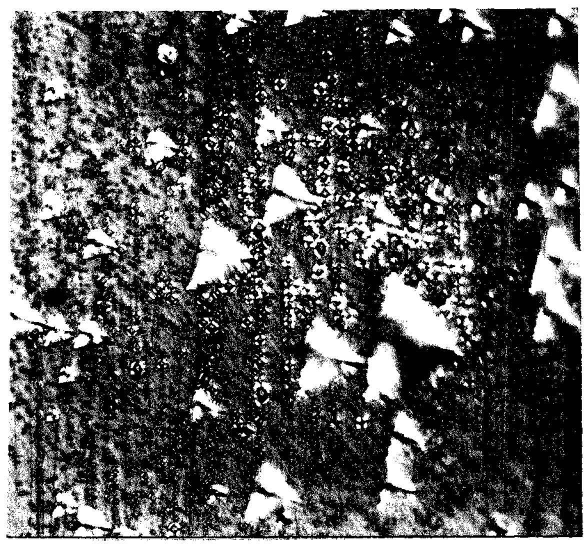

Figure 30
Same single crystal etched a third time using a different etchant (150 gms. $CuSO_4 \cdot 5\ H_2O$, 27 cc H_2SO_4, 50 cc Ethyl Alcohol and 1000 cc of H_2O) to show finer structure within the Lacombe subgrain. Glide polygonization is prevalent. 500 X, special tint plate.

V. SUMMARY

Of prime importance is the effect of glide strain on room temperature recovery. The greater the value of glide strain, the earlier subspots or fragments will appear in Laue back reflection photographs of stressed specimens left to recover at room temperature. It takes 6 days of room temperature recovery for the subspots to become evident when the applied glide strain is a minimum (0.073 for the E series). With increasing values of glide strain, the time gap for recovery is steadily decreased. A little less than a full day (18 hours) is required for a slightly greater value of glide strain (0.086 for the B series). Finally, subspots appear immediately after deformation for glide strain values of 0.175 and 0.209, corresponding to the F and G series respectively.

The shape, number, and internal structure of the Laue subspots are important evidence in revealing the nature of plastic deformation and the specific mode of room temperature recovery. The lower values of glide strain (E and B series) are associated with sharply defined subspots of almost equal spacing, and with a maximum of 3 subspots per original Laue spot. This uniformity of the Laue subspots indicates that plastic deformation is not complex in this case, having affected the metallic lattice in a very regular manner. If the lattice is bent, this bending is very slight, being concentrated in the subboundary area, a region of marked plastic curvature. In effect, the straight subboundaries which so clearly define the Laue fragments of Series E and B can be considered as areas where the concentration of excess edge dislocations - for simplicity - of one sign has been successfully assembled into polygon walls or transitional subboundaries. The main fragments or subspots can then be considered as regions of low dislocation density where the lattice retains a certain degree of perfection.

Room temperature recovery is evidenced through Laue photographs by a change in the relative intensity of the subspots with time. These changes are accompanied by subboundary movements, as the subboundaries themselves become increasingly sharp and better defined with time. These phenomena are particularly observable in the B series, taking place within 65 hours after stressing. The subspots tend towards a uniform intensity, that of the most intense subspot present. It appears that the subspots become more intense as the degree of internal perfection of the lattice structures which they represent increases, which is to say as they become depleted of dislocations. The small number of dislocations present in the main fragments would migrate towards the subboundary, there only to be annihilated by dislocations of opposite sign, and in the process helping the excess edge dislocations of one sign assume low energy configurations or polygon walls.

Thus, the main fragments and subboundaries tend to achieve perfection in a complementary fashion.

A consolidation in the number of subspots accompanies intensity changes which have been discussed in the preceding paragraph. Thus, after 65 hours at room temperature it is difficult to find more than 2 subspots per original Laue spot, as recorded in photograph B4 (Fig. 10). The Laue transmission method used on the L series has in turn presented evidence that entire subspots can disappear during room temperature recovery. The above observations are evidence that some of the structures in the Laue spots are transitory in nature, metastable with time, and that the deformed metallic lattice undergoes reversible changes in its search for a lower energy configuration.

Not all the subspots of the B series are sharp and devoid of a finer internal structure. In photographs B3 (Fig. 9) and B4 (Fig. 10), it was seen that two equal subspots are joined by a diffuse area, and that as the 2 outer subspots strive towards equal intensity, the central diffuse area becomes well defined, assuming a finer structure. These subspots correspond to lattice planes which have undergone plastic deformation in a more complex manner. It appears that the corresponding lattice planes have been severely bent and twisted in such a manner that the central area has become in effect a serious obstacle to the kind of dislocation movement which could bring about a uniform degree of recovery. This interpretation is backed by the observation that the central diffuse area fails to attain with time the degree of intensity of the two outer subspots.

Minute shifts in the X-ray beam indicate that room temperature recovery proceeds at different rates in different areas in the single crystal specimen. This is to be expected for the single crystal has undergone inhomogeneous deformation. The effect of glide strain on the speed of room temperature recovery is further determined and limited by the specific area of specimen studied. So, actually, the new area of specimen studied--due to a slight shift in the X-ray beam during the B series--did recover at a slower rate than would have been expected strictly on the basis of glide strain considerations. Fortunately, this observation cannot carry too much weight as an exception to the rule, since prior to the beam shift, the new area of Specimen B was not under observation, and it is quite possible that sharp subspots had appeared earlier, only to be masked by structural realignments of the deformed lattice. Nevertheless, the mode of recovery remains invariant, and the same structural changes accompanying recovery are experienced by neighboring areas, even though the recovery rates may be quite different.

Coarse fragments, irregular in shape, and limited by very poorly defined boundaries testify to the complex mode of plastic deformation exhibited by the F series. Contrasting with the subspots of Series E and B, these coarse fragment pairs are unequal in size, have no straight boundaries, and show a finer structure under the magnifying glass. The greater value of glide strain for the series (0.175) can be tied to the structural complexity of the Laue subspots. After one day at room temperature, changes in the relative intensity of the two subspots take place whereas the least intense subspot evolves into the more intense of the pair. Here again, these variations underline the metastability of the cold worked state. It is very likely that the excess dislocations produced by such complex deformation do not arrange themselves into large plane walls, as was postulated for the E and B series, but rather form a network of subboundaries which would tend to oppose any further dislocation rearrangements with time.

Immediately after deformation, Photograph G2 (Fig. 19) shows smeared Laue spots broken up into a great many fragments or subspots, a few of them relatively sharp. These spots have been smeared only in the direction of zone lines, appearing as narrow streaks, thus testifying to the directionality of plastic deformation in this instance. The deformation has not been as complex as in the F series, where coarse subspots were found to be extended in more than one direction.

As was expected, further recovery occured by holding at room temperature a control segment of specimen G.

However, a still greater amount of recovery was evidenced by the segment kept within the reactor for a period of 5 hours at 10 KW. For the irradiated segment, the effect of fast neutron damage was to replace the irregular structure within the Laue spot by sharp pairs of fragments of similar relative inclinations. The observed parallelism of these fragments could have been brought about by changes in the mutual configuration of the subspots due to vacancy annihilation on their boundaries. Szmid and Szarras[24] explained the relative twist of mosaic blocks after neutron irradiation on the same premises. Leighly, Perkins and McCune[4] suggested that excess vacancies introduced by neutron bombardment could be spent in assisting the process of dislocation climb to form low angle polygon boundaries. The sharp fragments observed after irradiation are very similar to the regular assemblies of E and B series. Bearing in mind the earlier advanced mechanism of recovery for the E and B series--migration of dislocations to the subboundaries to be incorporated into plane walls or polygon walls--the above interpretations are indeed quite applicable.

The effects of a low temperature anneal after the stressed specimen was held for a little longer than 2 days at room temperature were investigated. Identical corners were clipped from each segment to induce recrystallization, and both segments received a subsequent anneal at 330°C for a period of an hour. This was done to verify Guinier and Tennevin[8]'s experimental statement that up to a temperature of 450°C annealing leads to no perceptible alteration in the focused Laue spot. But, contrary to their expectations, and in spite of partial recovery of the segments at room temperature, further changes in the Laue spots were evidenced. However, the irradiated segment recovered to a greater extent than the control segment during the low temperature anneal. This conclusion was reached after examination of the structures within the "comet streaks" in Laue photographs taken of both segments after annealing. No evidence of recrystallization from the clipped corners appeared after etching both segments with Tucker's etchant. The experimental evidence suggests that increased recovery time at room temperature hinders recrystallization, in agreement with the observations by Leighly et al[30]. It is possible, as suggested by Wright[23], that room temperature recovery increases the recrystallization temperature. Further work in this area is needed in order to settle this question.

A new experimental approach using the Laue transmission on aluminum single crystals of foil thickness was tried, to circumvent the possibility of accidentally moving the experimental set up. Using this method, whose possibilities and limitations have been dwelt upon in Section IV, it was found that room temperature recovery produced alterations within the Laue subspots, and even the total disappearance of certain subspots. It is of interest to note that these changes were not gradual, but occured all at once upon the fifth day of recovery. Continued observation, up until the twenty fifth day after stressing, showed no further changes in the subspots.

The "double etch" method using Lacombe and Beaujard[21]'s reagent as modified by Gervais, Norton and Grant[17] was used in attempting to follow motion of individual dislocations, and especially of subboundaries during recovery at room temperature. Etch pits correspond to dislocations, usually in a one to one ratio, and are produced because the crystal is most highly strained where dislocations meet its surface, and there it is most easily attacked by the etchant. The "double etch" method is based on an etch-stress-etch sequence, and relies on observation of the bottom of etch pits to ascertain dislocation motion. Flat bottoms correspond to moved dislocations, and sharp bottoms to stable ones, for the etchant will widen any pre-existing pit, but will only continue to deepen it as long as the dislocation responsible for it remains. Observation of the bottom of these etch pits was made difficult by the high dislocation densities found in the original single crystals grown by the strain anneal method, and by the high reflectivity of the specimen surface. However, it was possible to observe the internal detail within the etch pits by swabbing the specimen surface with ink; but no evidence of subboundary movement during room temperature recovery appeared with successive etchings. The method, evidently has only limited applicability, for evidence of subboundary movements has been provided by the preceding X-ray studies. Either dislocation pinning inhibits movement at the surface, or the Lacombe etch pit structures do not reflect the complex rearrangements of the deformed metallic lattice during room temperature recovery. Some evidence of the latter alternative is shown in Figure 30 where etching with a different reagent has revealed the presence of a finer structure of glide polygons within the Lacombe subgrain.

VI. CONCLUSIONS

1) The degree of deformation of a single crystal, as given by its glide strain, exerts a sizable influence on the process of room temperature recovery. It determines the speed of recovery from plastic deformation; the greater the glide strain, the faster the appearance of fragments or subspots in the Laue photographs.

2) Room temperature recovery is evidenced through Laue photographs by a change in the relative intensity of the subspots with time. The subspots tend to consolidate in number, which means that some of the structures in the Laue spots are transitory in nature, metastable with time.

3) These changes are accompanied by subboundary movements, as the subboundaries themselves become increasingly sharp and defined with time. However, equilibrium is reached within a few days after stressing, and no further structural changes are subsequently observed.

4) Minute shifts in the X-ray beam indicate that room temperature recovery proceeds at different rates in different areas in the single crystal. This is to be expected for the single crystal will undergo inhomogeneous deformation and the appearance of the Laue subspots is different from point to point in the same specimen. Notwithstanding, the mode of recovery remains invariant, and the same structural changes accompanying recovery are experienced by neighboring

areas.

5) On the basis of a superficial investigation, recovery was found to be accelerated by fast neutron bombardment, as expected.

6) The experimental evidence indicates that increased recovery time at room temperature prevents recrystallization when the specimen undergoes a low temperature (330°C) anneal.

7) Metallographic evidence suggests that a process akin to polygonization is responsible for the limited amount of recovery at room temperature. The fact that polygonization is usually associated with "high temperature" recovery imposes limitations upon this mechanism for "low temperature" stress relief.

The limits imposed by the experimental method employed were all too evident at the close of this investigation; the conclusions in this area are as follows:

8) For the strain anneal method it was found that annealing temperature and thickness of starting material were the two most critical variables involved.

9) The Laue back reflection method best revealed the fine detail within Laue subspots. However, the possibility of accidentally moving the set up while positioning and removing the film holder is inherent to the method.

10) A new method using Laue transmission through thin single crystals of foil thickness was introduced for greater accuracy of results. This method is a very promising one; it drastically reduces the possibility of moving the experimental set up.

11) The "double etch" method was found to have limited applicability in the study of substructures. The bottom of etch pits was revealed through inking in order to determine the mobility of individual dislocations. However, no evidence of major subboundary movements during room temperature recovery resulted from observation of the interior of Lacombe etch pits.

12) It seems quite probable that the finer structure within the Laue subspots (striations, etc.) corresponds to etch structures within the Lacombe subgrains, as shown in Figure 30. It is, nonetheless, experimentally impossible to make a direct correlation.

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